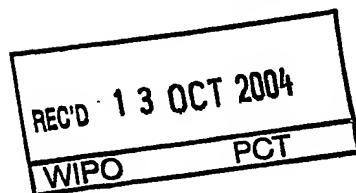




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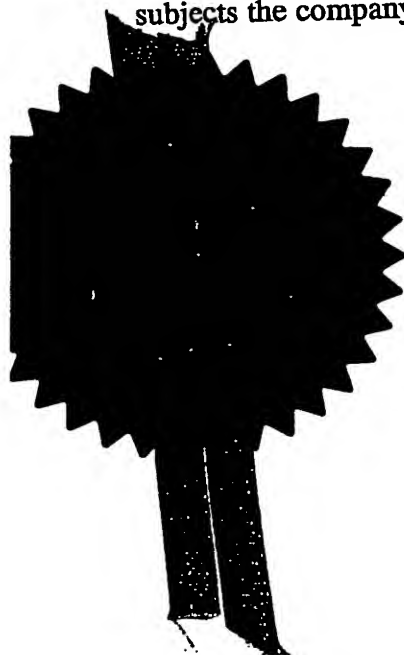
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2. Patent application number
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30 SEP 2003

0322907.7

3. Full name, address and postcode of the or of each applicant (underline all surnames)

Arjo Wiggins Limited

St. Clement House
Alencon Link, Basingstoke,
Hampshire, RG21 7SB

Patents ADP number (if you know it)

If the applicant is a corporate body, give the country/state of its incorporation

Great Britain

745553002

4. Title of the invention Improvements in Thermal Paper

5. Name of your agent (if you have one)

Abel & Imray

"Address for service" in the United Kingdom to which all correspondence should be sent (including the postcode)

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London
WC1R 4PQ

Patents ADP number (if you know it)

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Signature(s)

Abdel Imray

Abel & Imray

Date 30 Sept 2003

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Improvements in Thermal Paper

5

The present invention relates to thermal paper of the type in which print is developed by the direct application of heat to a thermally sensitive material.

- 10 In the manufacture of conventional thermal paper, a substrate such as paper is coated over its entire surface with an aqueous dispersion comprising colour formers, colour developers and sensitizers which are initially colourless but which become coloured on exposure to heat. Such dispersions
- 15 can often contain a wax, for example a stearamide wax. When such a paper is passed under the print head of a thermal printer, the areas which are activated by the heated print elements of the printer form coloured images on the surface of the paper. Such papers work well and produce clear
- 20 images, but are associated with a number of problems. The high chemical loading has been associated with environmental problems. The application of the thermal coating to the paper is an expensive operation, which must be carried out using complex and expensive coating equipment. And
- 25 crucially, conventional printing to thermal coating is difficult, and can only be carried out by performing an expensive surface treatment to achieve compatibility between ink and coating or by specific printing processes e.g. UV cured inks.

30

Attempts have been made to develop a thermal ink which reduces the problems associated with thermal papers by obviating the need to provide a thermal coating over the whole surface of the paper. US 5,888,283 describes a thermal

ink which can be printed onto paper using conventional printing processes, thus eliminating the need to use coating equipment. The ink is pigment free, and comprises an aqueous dispersion of an initially colourless colour former and an initially colourless colour developer which combine to form colour upon the application of heat, the ink having a solids content of at least 40% by weight. It preferably includes a sensitizer which at least partially surrounds the particles of colour developer. Suitable sensitizers include diphenoxyethane, aryl or alkyl-substituted biphenyls such as p-benzyl biphenyl, or toluidide phenyl hydroxynaphthoates and aromatic diesters such as dimethyl or dibenzyl terephthalate and dibenzyl oxalate. These materials may be used alone, or they may be combined with waxes or fatty acids. The ink is applied by a flexographic or gravure printing process, and develops colour when passed through a direct thermal printer.

The development described above has, however, a number of disadvantages. Specifically, using standard "stock" substrates under standard thermal imaging equipment leads to poor image formation. High energy laser sources are needed to produce acceptable image intensity. EP 600 441A describes a method which comprises irradiating a printed surface with a laser, the surface being printed with an ink which comprises a leuco dye as a colour former, an acidic substance as a colour developer, and at least one background colour formation inhibitor which is a water-soluble amino acid, and ammonium salt of an inorganic acid, a pH buffer, or water. However, laser printer types are not standard within the thermal paper industry and require cost expenditure to replace traditional thermal printing equipment if they are to be used.

There is therefore a need for a system in which a thermal ink can be used to provide good print quality using standard thermal imaging equipment, there is minimal discolouration during storage, and the product can be readily and economically printed using conventional non thermal imaging processes before it is thermally printed.

We have developed a thermal printing ink which, when printed on a very specific substrate, is resistant to premature colouration during storage of the coated papers, and may be imaged to produce high quality prints using standard thermal writing equipment at standard energy levels. Because the present invention uses a thermal ink rather than a thermal coating as used in conventional thermal paper, the ink can be applied by printing on specific areas of a sheet. This reduces chemical costs and also allows the non-coated areas on the sheet to be printed by conventional means (wet offset and the like) to add value to the sheet, which is generally not possible using conventional thermal papers. The printing of the thermal ink can be carried out at the same time as the printing of information using conventional ink, which means that, for the first time, it is possible to produce visually attractive products such as labels, tickets or till rolls which carry high quality conventionally printed information, which will develop a high quality thermal image when subsequently passed through a thermal printer. The substrate used to carry the printed material may be made on a conventional paper making machine and, unlike conventional thermal coated papers, does not require subsequent processing using a separate coating machine.

Accordingly the present invention provides a thermal ink which comprises a colour former, a colour developer and a sensitizer, characterised in that the sensitizer is dimethyl

terephthalate, and that the ink also comprises at least one pigment.

Preferably the thermal ink of the present invention is free from wax. In conventional thermal formulations, wax is used to reduce unwanted discolouration of the thermal paper during storage. It is a surprising feature of the present invention that discolouration can be reduced without the presence of wax in the formulation.

10

Any suitable colour former and colour developer may be used in the ink of the present invention. Suitable colour formers include, for example, diaryl methanes including 4,4-bis(dimethylaminobenzyldroxybenzyl)ether, N-halophenyl, leuco auramine, and N-2,4,5-trichlorophenyl leuco auramine; fluorans including 2-dibenzylamino-6-diethylaminofluoran, 2-anilino-6-diethylaminofluoran, 3-methyl-2-anilino-6-diethylaminofluoran, 2-anilino-3-methyl-6-(ethylisopentylamino)fluoran, 2-anilino-3-methyl-6-butyl aminofluoran, 2-chloro-3-methyl-6-diethylaminofluoran, 3,6-dimethoxyfluoran, and 7,7'-bis(3-diethylaminofluoran); spiropyrans including 3-methylspirodinaphthopyran, 3-ethylspirodinaphthopyran, 3,3'-dichlorospirodinaphthopyran, 3-benzyl spironaphthopyran, and 3-methylnaphtho-(3-methoxybenzo)spiropyran; azaphthalides including 3-(2-ethoxy-4-diethylaminophenyl)-3-(1-octyl-2-methylindol-3-yl)-4-azaphthalide, and 3-(2-ethoxy-4-diethylaminophenyl)-3-(1-ethyl-2-methylindol-3-yl)-4-azaphthalide; indolylphthalides including 3-(p-dimethylaminophenyl)-3-(1,2-dimethylindol-3-yl)phthalide and 3-(p-dimethylaminophenyl)-3-(2-methylindol-3-yl)phthalide; thianyl methanes; and styryl quinoline.

25

30

Preferred colour formers for use in the present invention are 3,3-bis(4-dimethylaminophenyl)-6-dimethylaminophthalide.

- common name CVL (blue); 2'-(dibenzylamino)-6'-(diethylamino)spiro(isobenzofuran-1(3H),9'-xanthen)-3-one, Fuji Green; spiro[isobenzofuran-1(3H),9'-[9H]xanthen]-3-one,6'-(diethylamino)-2'-octylamino-, ODB1 (black); 3-dibutylamino-6-methyl-7-anilinofluoran (alternative nomenclature: spiro[isobenzofuran-1(3H),9'[9H]xanthen]-3-one,-6'(dibutylamino)-3'-methyl-2'-(phenylamino)-, ODB2 (black); 2'-anilino-6'-(ethyl(isopentyl)amino)-3'-methylspiro(isobenzofuran-1(3H),9-xanthen)3-one, S-205 (black); 3-diethylamino-6-methyl-7-(3'-methylanilino)fluoran, ODB7 (black); benzenamine,4,4'((9-butyl-9H-carbazol-3-yl)methylene)bis(N-methyl-N-phenyl-), SRB (blue); 6'-diethylamino-3'-methyl-2'-(2,4-xylidino)spiro(isobenzofuran-1(3H),9-xanthen)3-one, Black XV; 2-methyl-6-(N-p-tolyl-N-ethylamino)-fluoran, ETPM (red); spiro(isobenzofuran-1(3H),9'-(9H)xanthen)-3-one,3'-chloro-6'-(cyclohexylamino), Orange 100; 3,3'-bis(2-methyl-1-octyl-1H-indol-3-yl)-1(3H)-isobenzofuranone, Red 1-6B; Orange 1-G; Red MC30; Yellow 1-3R; and 3,3-bis(2,2-bis(4-(dimethylamino)phenyl)ethenyl)-4,5,6,7-tetrachloro-1(3H)-Isobenzofuranone, a green/black with activity in the near infrared useful in optical character recognition applications (OCR), trade name MG1 (Marks Chemicals).
- 25 A particularly preferred colour former for use in the present invention which has demonstrated particular advantages is 3-dibutylamino-6-methyl-7-anilinofluoran (alternative nomenclature: spiro[isobenzofuran-1(3H),9'-[9H]xanthen]-3-one,-6'-(dibutylamino)-3'-methyl-2'-(phenylamino)-, known by 30 the common name ODB2, CAS number 89331-94-2, and available under the Trade Marks Black 1-2R (Ciba), Black T-2R (Ciba), and PSD 184 (Nisso).

Suitable colour developers include, for example, bis-(3-allyl-4-hydroxy phenyl)sulphone, 2,4-dihydroxy diphenyl sulphone, p-hydroxybenzylphenol, 4,4'-disulphonyl phenol, 3-benzyl salicylic acid, 3,5-di-t-butylsalicylic acid, 4-hydroxyphenyl-4-isopropoxyphenylsulphone, 4,4'-thiodiphenol phenol-formaldehyde novolac resin, alphanaphthol, bisphenol A, bisphenol sulphone, benzyl 4-hydroxybenzoate, 3,5-dimethyl-4-hydroxybenzoic acid, 3-isopropylsalicylic acid, 4,4'-isopropylidene diphenol, and 3,3'-dimethyl-4,4'-thiodiphenol.

A particularly preferred colour developer for the present invention is bisphenol A.

The pigment comprised in the thermal ink according to the invention is preferably a high surface area, absorptive pigment, for example precipitated calcium carbonate, silica or calcined clay. Surprisingly, the presence of a pigment does not render the ink unsuitable for use in the intended application; rather, the pigment helps to prevent migration of the ink onto the thermal printing head during imaging, giving a high quality image.

The ink suitably consists of three individual components mixed to form the ink precursor:

- 1) A dye system which includes the colour former and DMT, and which may also include components such as, for example, one or more surfactants, preferably a polyvinyl alcohol surfactant and optionally additional surfactants, and antifoam agents.
- 2) A coreactant system which includes the colour developer and DMT, and which may also include components such as, for example, one or more surfactants, preferably a

polyvinyl alcohol surfactant and optionally additional surfactants, and antifoam agents.

3) A pigment dispersion in slurry form.

- 5 The thermal ink of the present invention is suitably prepared by grinding the above three components separately. These separate grinding operations reduce the tendency for unwanted colour reaction and produce a non-coloured ink vehicle. Preferably the particles in each component are ground to a
- 10 particle size of less than 1.5μ , especially less than 1.0μ , for example from 0.25μ to 1.0μ . The components are then mixed together, optionally including additional components such as slip agents and defoamers, to form the resulting ink, which may be printed using conventional means, being
- 15 compatible with standard flexographic printing processes in terms of viscosity and cell transfer. Ink in which the solid particles have a particle size of less than 1.5μ produces especially advantageous results.
- 20 The thermal ink according to the invention has particularly useful properties when used in combination with a particular substrate. In this substrate, a sheet, which may for example be synthetic paper or polymer film but which is preferably paper, is coated on at least one surface with a coating
- 25 comprising a layer containing a pigment in solid porous particulate form. Accordingly, the invention also provides a thermally printable sheet which comprises a base sheet having at least one surface coated with a layer containing a pigment in solid porous particulate form, and printed upon
- 30 said coated surface, a thermal ink according to the invention. The pigment present in the coating has a high surface area and a high absorptivity, preferably with a surface area measured at $>100 \text{ m}^2/\text{g}$ using the BET method or an ink absorbency of $>50\text{g oil}/100\text{g pigment}$ (as described in

Kirk-Othmer Encyclopedia of Chemical Technology, 3rd Edition, Volume 17, pages 796 - 808). It preferably comprises calcined clay, calcium carbonate (in precipitated form, which is porous and of high absorptivity), and/or silica. The
5 coating also preferably comprises at least one additional pigment, preferably a plastic pigment in the form of hollow spheres.

The thermally printable sheet of the present invention may be
10 developed using conventional thermal printers. In such printers, heat is generated by the application of short pulses of low-power electrical energy. There is no requirement to use specialist equipment such as lasers.

15 The thermally printable sheet according to the invention may be prepared by printing a thermal ink according to the invention onto the coated surface of the substrate, using a printer. It is a major advantage provided by the invention that the coating applied to the substrate may be applied to
20 the whole surface of a sheet of paper during a conventional paper-making process without the use of separate, expensive coating machines, while the thermal ink can be printed onto only those parts of the coated surface where thermal activity is required, using a conventional printer, if desired at the
25 same time as conventional ink is printed onto the surface to provide high-quality visible information.

The specific coating present on the substrate provides a number of advantages when used in combination with the
30 thermal ink according to the invention. The thermal ink is held near the surface of the sheet to permit good contact with the thermal printing head and maximise print formation, and there is adequate absorption of the thermal ink to prevent unwanted contamination of the thermal printing head

which impairs print quality. In addition, thermal insulation is provided such that energy applied to the surface is maximised within a localised area rather than being dissipated through the sheet. This maximises the thermal
5 print formation.

Most surprisingly, the use of the thermal ink according to the invention in combination with a substrate bearing a coating comprising a layer containing a pigment in solid
10 porous particulate form, leads to a significant reduction in discolouration during storage compared with conventional thermal papers. Accordingly the invention provides the use of DMT in a thermal ink comprising a colour former and a colour developer, to reduce unwanted discolouration during
15 storage of a thermally printable sheet product comprising a base sheet having at least one surface coated with a layer containing a pigment in solid porous particulate form, absorptive pigment, said thermal ink being printed upon said coated surface. The invention further provides the use of a
20 combination of a thermal ink comprising DMT, a colour former and a colour developer, and a surface coating comprising a layer containing a pigment in solid porous particulate form, to reduce unwanted discolouration during storage of a thermally printable sheet product.

25

The following Example illustrates the invention.

Example

30 Dye Material

Under agitation of <100 rpm using a Greaves or Silverson mixer, 2.25 dry parts of a 20% Polyvinyl alcohol solution (Moviol 4/88 - Clariant or Poval 203 - Kurraray) was added to

a pre-selected quantity of water to achieve 40% total solids of the dye material blend. 0.08 dry parts of Surfinol 420 (Air Products) were quickly added to reduce foaming.

Agitation was increased to 200 rpm and 4.56 dry parts of
5 colour former, such as Pergascript T2R (Ciba Specialty Chemicals) were added, and mixing continued until the colour former was fully dispersed. 2.58 dry parts of DMT sensitiser (Molekula Ltd., UK) were then added to the mixture under agitation of <100 rpm. Lastly a very small amount of Drewplus
10 S4386 defoamer (Drew) was added.

The resulting fully dispersed mixture was added to a bead mill and continued to be ground until a size of less than 1 μ m median size and preferably \pm 0.5 μ m median size was achieved, as measured using a Malvern Multisizer. Particular attention
15 was taken to ensure that the temperature during the grinding process did not rise to above 30°C. This prevented unwanted gellation of the mixture.

Co-reactant Material

20

Under agitation of <100 rpm using a Greaves or Silverson mixer, 2.25 dry parts of a 20% Polyvinyl alcohol solution (Moviol 4/88 - Clariant or Poval 203 - Kurraray) was added to a pre-selected quantity of water to achieve 40% total solids
25 of the co-reactant material blend. 0.08 dry parts of Surfinol 420 (Air Products) were quickly added to reduce foaming. Agitation was increased to 200 rpm and 10 dry parts of Bisphenol A coreactant (Molekula Ltd., UK) were added. Mixing continued until the colour former was fully dispersed.
30 2.58 dry parts of DMT sensitiser (Molekula Ltd., UK) were then added to the mixture under agitation of <100 rpm. Lastly a very small amount of Drewplus S4386 defoamer (Drew) was added.

The resulting fully dispersed mixture was added to a bead mill and continued to be ground until a size of less than 1 μ m median size and preferably \pm 0.6 μ m median size was achieved, as measured using a Malvern Multisizer. Particular attention
5 was taken to ensure that the temperature during the grinding process did not rise to above 30°C. This prevented unwanted gellation of the mixture.

Pigment Material

10

Commercial high surface area pigment such as precipitated calcium carbonate (Calopake - F, SMI) was added to a pre-selected quantity of water to achieve a final solids of 55%. The mixture was agitated using a Greaves or Silverson mixer
15 such that a particle size of less than 1.5 μ m median size was achieved.

Ink Mixture

- 20 In a large stirred beaker or similar vessel, a pre-selected quantity of water to achieve a final ink solids of 46% was added followed by the co-reactant material mixture. 5.7% dry parts of a 30% solution of Hidorin Z-7-30 (Chukyo Europe) was added to the stirred co-reactant mixture.
- 25 The pigment material blend was next added to the stirred vessel and lastly the dye material was added.

The composition of the resultant ink is shown in the following Table.

Material	% by weight
Polyvinyl alcohol - Moviol 4/88	4.5
Bisphenol A	10
Black T2R	4.56
Dimethyl terephthalate	5.7
Calopake-F	20
Zinc stearate - Hidorin Z-7-30 (30%E.E)	5.7
Surfinol 420	0.3
Drew N40 (defoamer)	0.1
Total	100

- 5 The ink was applied by a laboratory flexographic printing unit at a coatweight range of up to 4gsm. Using a conventional thermal fax printer, at a pulse signal range of 1.0 - 1.4msec images were produced, and the results are given in Table 2. For comparison, the experiment was repeated
- 10 using an uncoated base paper. Higher numbers obtained using a Gretag reader indicate a higher visibility printed image.

	Gretag Measurement	
	1.2msec	1.4msec
Uncoated base paper	0.06	0.04
Precoated base paper	0.31	0.72

- 15 It can be seen that the print quality is significantly better using the coated paper than using the uncoated paper. In both cases, minimal transfer of ink to the thermal printer head was observed.

Claims:

5

1. A thermal ink which comprises a colour former, a colour developer and a sensitizer, characterised in that the sensitizer is dimethyl terephthalate, and that the ink also comprises at least one pigment.

10

2. A thermal ink as claimed in claim 1, in which the colour former is 3-dibutylamino-6-methyl-7-anilinofluoran.

15

3. A thermal ink according to either claim 1 or claim 2, which is free of wax.

20

4. A thermal ink according to any one of claims 1 to 3, in which the pigment is calcined clay, precipitated calcium carbonate, and/or silica.

25

5. A thermal ink according to any one of claims 1 to 4, in which the particle size of solids present in the ink is less than 1.5μ , preferably less than 1.0μ .

6. A thermal ink according to any one of claims 1 to 5, in which the colour developer is bisphenol A.

30

7. A thermal ink according to any one of claims 1 to 6, which also comprises polyvinyl alcohol.

8. A method of preparing a thermal ink according to any one of claims 1 to 7, which comprises grinding together the colour former and DMT; in a separate operation, grinding together the colour developer and DMT; and subsequently

blending together said ground products together with a pigment.

9. A thermally printable sheet which comprises a base sheet
5 having at least one surface coated with a layer containing a pigment in solid porous particulate form, there being printed upon said coated surface, a thermal ink as claimed in any one of claims 1 to 7.
- 10 10. A thermally printable sheet as claimed in claim 9, in which said pigment is calcined clay, precipitated calcium carbonate and/or silica.
11. A thermally printable sheet as claimed in either claim 9
15 or claim 10, in which the surface coating also comprises at least one additional pigment.
12. A thermally printable sheet as claimed in claim 11, in
20 which said additional pigment is a plastic pigment in the form of hollow spheres.
13. A method of preparing a thermally printable sheet as claimed in any one of claims 9 to 12, which comprises
25 printing a thermal ink as claimed in any one of claims 1 to 7 onto the coated surface of a substrate which is a sheet having at least one surface coated with a layer containing a pigment in solid porous particulate form, using a printer.
14. The use of DMT in a thermal ink comprising a colour
30 former and a colour developer, to reduce unwanted discolouration during storage of a thermally printable sheet product comprising a base sheet having at least one surface coated with a layer containing a pigment in solid porous

particulate form, said thermal ink being printed upon said coated surface.

15. The use of a combination of a thermal ink comprising
5 DMT, a colour former and a colour developer, and a surface coating comprising a pigment in solid porous particulate form, to reduce unwanted discolouration during storage of a thermally printable sheet product.

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